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THE MOLECULAR STRUCTURE OF BIS(DIMETHYLAMINO)-DIFLUOROSULFURANE--ETC(U)

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The molecular structure of $(Me_2N)_2SF_2$ has been determined by single crystal X-ray diffraction methods. The compound crystallizes in the monoclinic system, space group C2/c, with a = 11.00 (2) Å, b = 5.693 (6) Å, c = 12.24 (3) Å, β = 92.79 (10)°, and Z = 4. The symmetry of $(Me_2N)_2SF_2$ is C_2, and the structure is essentially trigonal bipyramidal with the fluorine and Me_2N ligands occupying axial and equatorial sites,

beta

deg

respectively. The third equatorial site is occupied by the sulfur "lone pair" which lies along the C_2 symmetry axis. Since the dihedral angle between the C-N-C and N-S-N planes is 48.2°, the nitrogen lone pairs are disposed approximately midway between the sulfur lone pair and the sulfur-fluorine axial ($S-F_2$) bonds. The sum of the bond angles around the nitrogen atoms is 342.3°; hence the hybridization at these centers is approximately halfway between sp^2 and sp^3. The F_a-S-F_a bonds are bent toward the sulfur lone pair by 5.3°, and the equatorial N-S-N bond angle is 102.3(1)°. The S-F and S-N bond distances are 1.770(2) Å and 1.648(2) Å, respectively.

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THE MOLECULAR STRUCTURE OF BIS(DIMETHYLAMINO)-DIFLUOROSULFURANE; THE FIRST X-RAY CRYSTAL STRUCTURE OF A FLUOROSULFURANE

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Abstract: The molecular structure of $(Me_2N)_2SF_2$ has been determined by single crystal X-ray diffraction methods. The compound crystallizes in the monoclinic system, space group C2/c, with a = 11.00 (2) Å, b = 5.693 (6) Å, c = 12.24 (3) Å, β = 92.79 (10)°, and Z = 4. The symmetry of $(Me_2N)_2SF_2$ is C_2, and the structure is essentially trigonal bipyramidal with the fluorine and Me_2N ligands occupying axial and equatorial sites, respectively. The third equatorial site is occupied by the sulfur "lone pair" which lies along the C_2 symmetry axis. Since the dihedral angle between the C-N-C and N-S-N

planes is 48.2°, the nitrogen lone pairs are disposed approximately midway between the sulfur lone pair and the sulfur-fluorine axial (S-F_a) bonds. The sum of the bond angles around the nitrogen atoms is 342.3°; hence the hybridization at these centers is approximately halfway between sp^2 and sp^3. The F_a-S-F_a bonds are bent toward the sulfur lone pair by 5.3°, and the equatorial N-S-N bond angle is 102.3(1)°. The S-F and S-N bond distances are 1.770(2) Å and 1.648(2) Å, respectively.

INTRODUCTION

The sulfuranes, which feature an unshared pair of electrons and a coordination number of four at the sulfur atom, are examples of a broader category of species for which Musher[1] coined the phrase "hypervalent molecules." Bonding models for such molecules have been advanced and discussed for several years.[1,2] In recent times molecular orbital (MO) calculations have provided useful insights into the nature of the bonding in the model system, SH_4,[3] and the simplest known sulfurane, SF_4.[4]

Although X-ray crystallographic studies have been performed on several spirocyclic sulfuranes,[5-9] structural information on the acyclic sulfuranes is somewhat sparse. The molecular geometry of SF_4 has been well established on the basis of microwave spectroscopic[10] and electron diffraction[11] data, and X-ray crystallographic data are available for $(ClC_6H_4)_2SCl_2$[12] and $(C_6H_5)_2S[OC(CF_3)_2C_6H_5]_2$[13]. The present paper is concerned with an X-ray crystallographic investigation of $(Me_2N)_2SF_2$; as well as being the first fluorosulfurane to be studied by X-ray diffraction methods, the molecule is of special interest on account of the presence of three proximate lone pairs of electrons. As noted by Chen and Hoffmann[4a], the structures of R_2N-substituted sulfuranes "would be of great interest" because of the operation of two competing effects, viz. the tendency of the sulfur and nitrogen lone pairs to avoid each other, and the opposing tendency to maximize dative p-d π-bonding. Additional struc-

tural information on sulfuranes is also pertinent to further refinements of the theoretical models to assist in understanding site preferences and bond angle trends. Finally, we note that the structures of dialkylamino-substituted sulfuranes are of practical interest since these compounds are used as fluorinating agents[14] and as precursors to sulfonium cations.[15]

Experimental Section

The sample of $(Me_2N)_2SF_2$ was prepared according to the method of Middleton.[14c] In order to grow single crystals, small quantities of $(Me_2N)_2SF_2$ were sublimed into 30 capillaries, each of which was sealed off in vacuo. Sublimation was accomplished by heating one end of the capillary with a 60 watt light bulb, while maintaining the other end at ambient temperature. From these samples one satisfactory crystal was obtained. It was transferred in its capillary to a goniometer head and then to a Syntex $P2_1$ diffractometer. During the course of all subsequent crystallographic experiments the crystal and its capillary were maintained at -35°C by a stream of cold N_2. Preliminary X-ray diffraction experiments indicated the monoclinic symmetry of space group Cc (no. 9) or C2/c (no. 15). Crystal data and X-ray diffraction data collection details are summarized in Table I. Processing of the diffraction data (with p = 0.02) was carried out as described previously.[16]

Solution and Refinement of the Structure. A calculated

density of 1.38 g cm^{-3} (see Table I) is consistent with four molecules of $(Me_2N)_2SF_2$ per unit cell. Thus, while the molecules would have no crystallographically imposed symmetry in space group Cc, they would be required to reside at sites of either C_i or C_2 symmetry in space group C2/c. Solution of the structure by standard heavy atom methods and satisfactory refinement by full-matrix least-squares procedures showed the space group to be C2/c and revealed that the molecules occupy sites of C_2 symmetry. The function minimized in refinement is $\Sigma w(|F_o| - |F_c|)^2$, where the weight w is $\sigma(|F_o|)^{-2}$, the reciprocal square of the standard deviation of each observation, $|F_o|$. Neutral atom scattering factors for S, F, N, C[17] and H[18] were used in these calculations, and the real ($\Delta f'$) and imaginary ($\Delta f''$) corrections[17] for anomalous scattering were applied to the sulfur scattering curve.

Least-squares convergence was attained using only those 656 data with $I_o/\sigma(I_o) > 2.0$ for a structure in which non-hydrogen atoms were refined anisotropically and hydrogen atoms isotropically, with $R = \Sigma||F_o| - |F_c||/\Sigma|F_o| = 0.057$, $R_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2} = 0.043$ and a standard deviation of an observation of unit weight $= [\Sigma w(|F_o| - |F_c|)^2/(m-s)]^{1/2} = 1.56$, for m = 652 observations and s = 66 variables. **Examination** of the data near the conclusion of refinement revealed that four reflections (002, 110, 200, $11\bar{2}$) were apparently affected by secondary extinction. These reflections were deleted from the data set prior to final least-squares refinement.

In the final cycle of refinement all parameter shifts were less than 0.7% of a corresponding estimated standard deviation (esd) for nonhydrogen atoms and less than 1.7% of an esd for the hydrogen atoms. A final difference Fourier map showed no peak higher than 0.14 e $Å^{-3}$. For comparison, the heights of the carbon atoms from a previous Fourier map were 2.5 and 3.1 e $Å^{-3}$.

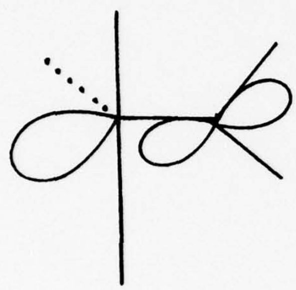
A listing of computer programs used in this work is provided elsewhere.[16] Atomic positional and thermal parameters with corresponding esd's as estimated from the least-squares inverse matrix are given in Table II. A tabulation of observed and calculated structure factor amplitudes is available.[19]

Discussion

The structure of $(Me_2N)_2SF_2$ can be described as essentially trigonal bipyramidal. A stereoview of the molecule and a view approximately down the C_2 symmetry axis are presented in Figures 1 and 2, respectively. The fact that both Me_2N groups occupy equatorial sites and both fluorine ligands occupy axial sites is anticipated on the basis of polarity rules[20] and confirms the structure suggested for this molecule from NMR spectroscopic evidence.[21] The third equatorial site can be considered to be occupied by the sulfur "lone pair" which is collinear with the C_2 axis.[22] Significant aspects of the structure of $(Me_2N)_2SF_2$ are now discussed.

1. The Conformations of the $Me_2\ddot{N}$ Groups. Particular

interest focuses on the nitrogen geometries and the stereochemical relationships between the Me_2N groups and the sulfur "lone pair". Chen and Hoffmann[4a] in their theoretical analysis of the model sulfurane, H_2NSH_3, have pointed out that dative π-bonding from filled N(2p) orbitals favors conformation 1, while repulsion between the nitrogen and sulfur lone pairs



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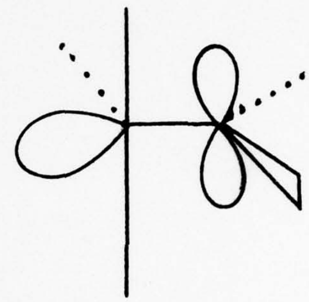
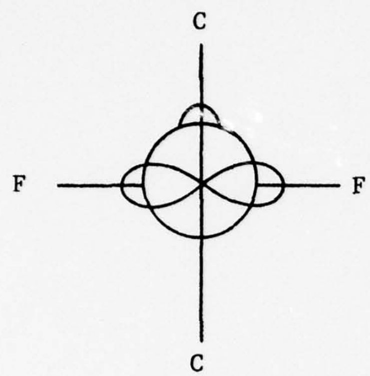


2

favors conformation 2. Interestingly, in the case of $(Me_2N)_2SF_2$ the Me_2N groups exhibit a dihedral angle approximately midway between 1 and 2 (Table III and Figure 2), presumably as a compromise between these factors. It is also noteworthy that, since the sum of the bond angles around nitrogen is 342.3°, the hybridization is approximately halfway between sp^2 and sp^3. This observation may be rationalized by appeal to our theoretical studies of other systems containing two or more lone pairs, such as the aminophosphines.[23] For example, in the case of H_2NPH_2 it was demonstrated that the nitrogen geometry is trigonal planar when the nitrogen and phosphorus lone pairs occupy orbitals

which are orthogonal, but approximately tetrahedral when the lone pairs are eclipsed. Applying these principles to $(Me_2N)_2SF_2$ one would anticipate a trigonal planar nitrogen geometry in conformation 3 and an approximately tetrahedral conformation in 4.



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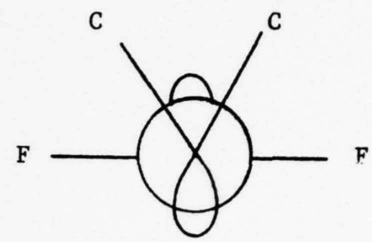

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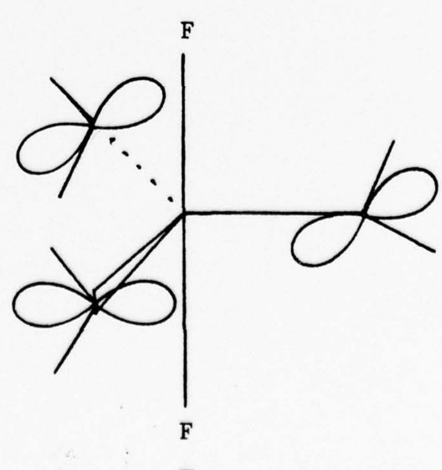
Clearly, the fact that $(Me_2N)_2SF_2$ adopts a conformation intermediate between 3 and 4 implies that the response of nitrogen geometry to inter-lone pair angle is monotonic. The deduction that the nitrogen geometry in $(Me_2N)_2SF_2$ is influenced by the sulfur lone pair rather than other structural features is supported by the observation that in the closely analogous fluorophosphorane, $(Me_2N)_3PF_2$, 5, the nitrogen geometries are,



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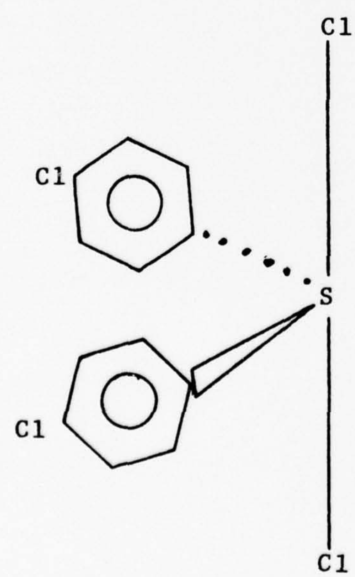
within experimental error, trigonal planar.[24]

The two shortest intermolecular methyl . . . methyl distances in this crystal structure are 3.571 (6) and 3.963 (6) Å. The van der Waals methyl . . . methyl contact distance is 4.00 A.[25] However, since all intermolecular H . . . H distances are greater than 2.60 Å and since the van der Waals H . . . H contact distance is ~2.0 A,[26] there are no methyl . . . methyl interactions in this crystal structure which have a significant effect upon the geometry of the Me_2N groups.

2. THE AXIAL (HYPERVALENT) BONDS. The fact that the F-S-F bonds are bent toward the sulfur lone pair (by 5.3°) is noteworthy. To our knowledge, the only other case where this occurs is in the chlorosulfurane, 6.[12] Since short



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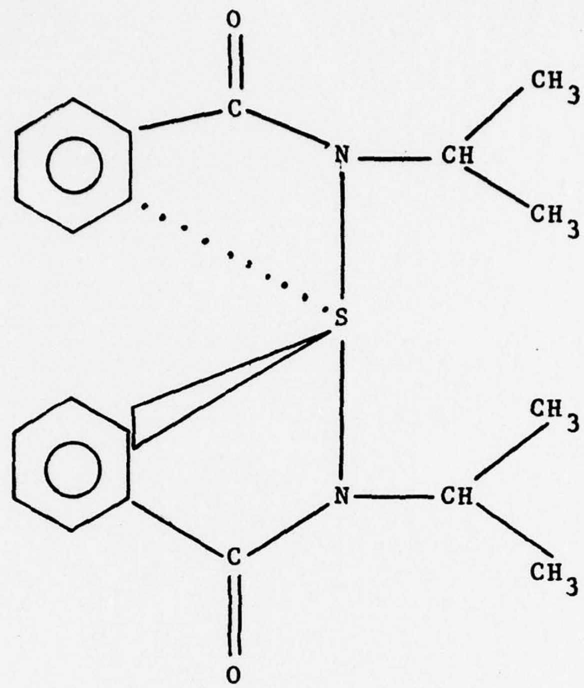
intermolecular S·····Cl and S-Cl·····Cl-S contacts were observed in the crystal structure, the distortion in 6 seems best attributed to crystal packing effects. That the bending of S-F bonds toward the lone pair of electrons of the sulfur atom in $(Me_2N)_2SF_2$ is also due to intermolecular interactions rather than electronic effects has been considered. Two rather short F···C contacts of 3.319 (5) and 3.524 (5) Å (from two neighboring molecules) occur, with corresponding F···H distances of 2.40 (4) and 2.56 (4) Å. These values may be compared to the sums of the van der Waals radii of 3.35 Å for F and C atoms,[25] and 2.35 Å for F and H atoms.[25,26] The C-H···F angles are 167(3) and 152 (2)° (close to linearity) and, as may be seen in Figure 3, these C-H···F interactions appear to be attractive rather than repulsive. Hence, the possibility that the small bending of the S-F bonds toward the lone pair of the sulfur atom may be due partially or wholly to weak attractive intermolecular forces cannot be dismissed.

From an electronic standpoint, it would be reasonable to ascribe the observed F_a-S-F_a angle to repulsions between the fluorine ligands and the nitrogen lone pairs. Alternatively, Chen and Hoffmann[4a] have presented a perturbational MO argument to the effect that at a fixed equatorial bond angle, θ_1, (see 7) the axial bond angle, θ_2, will increase with increasing disparity in electronegativity between the axial and equatorial ligands. This argument is consistent with the fact that for SF_4 θ_2 = 173.1°, while for $(Me_2N)_2SF_2$ θ_2 = 185.3°.



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It is clear that, while the relatively small θ_1 value for $(Me_2N)_2SF_2$ is understandable on this basis, the general correlation between θ_1 and the electronegativity of the atoms at axial positions is poor.

Finally, one other trend predicted by Chen and Hoffmann[4a] -- namely that θ_1 should decrease as the axial bond distances increase - is not borne out by the available data in Table IV.

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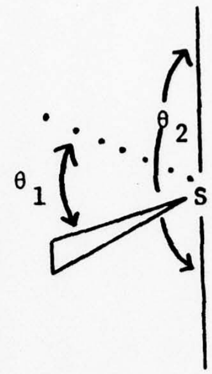

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3. THE EQUATORIAL BONDS. The S-N bond distances (equal by symmetry) for $(Me_2N)_2SF_2$ are 1.648 Å and thus substantially less than the sum of the covalent radii for sulfur and nitrogen (1.75 Å).[25] This is perhaps not surprising since the covalent radius for nitrogen is predicated on the assumption of tetrahedral geometry. Furthermore, expansion of the C-N-C angle is expected to impart more N(2s) character to the bonds, thereby shortening them. The only other crystallographically characterized sulfurane with S-N bonds is the interesting spirocyclic species, 8.[9] Obviously, here the nitrogen atoms are in axial positions and, as expected, these hypervalent S-N bonds are considerably longer (average, 1.898 Å) than the equatorial S-N bonds in $(Me_2N)_2SF_2$.

Chen and Hoffmann[4a] and Paul, Martin, and Perozzi[13] have independently suggested that, for sulfurane substitution by more electronegative ligands at the axial sites, a smaller equatorial bond angle, θ_1, is probable. The available structural data bearing on this point are summarized in Table IV.

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(24) The sum of angles at each nitrogen atom is 357.4 $\pm$ 2.4°. The dihedral angle between each CNC plane and the equatorial plane is 70.1 $\pm$ 2.7°. See H. Oberhammer and R. Schmutzler, *J. Chem. Soc. Dalton Trans.*, 1454 (1976). The conformation **5** was predicted earlier on the basis of UV photoelectron spectroscopic measurements. See A. H. Cowley, M. J. S. Dewar, D. W. Goodman, and J. R. Schweiger, *J. Am. Chem. Soc.*, **95**, 6506 (1973).

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Table I. Crystallographic Summary

Crystal Data at -35°C[a]

a, Å	11.00 (2)	Systematic absences	$hk\ell$, $h + k = 2n + 1$
b, Å	5.693 (6)		$h0\ell$, $\ell = 2n + 1$
c, Å	12.24 (3)		$0k0$, $k = 2n + 1$
β, deg	92.79 (10)		
V, Å^3	766 (6)	Crystal system	Monoclinic
Mw	158.22		
d_{calcd}[b] g cm^{-3}	1.38	Space group[c]	Cc (no. 9) or $C2/c$ (no. 15)
Z	4		
F(000), electrons	336	Empirical formula	$C_4H_{12}F_2N_2S$

Data Collection at -35°C[d]

Radiation (MoKα), Å	0.71069
Mode	ω scan
Scan range	Symmetrically over 1.25° about $K\alpha_{1,2}$ maximum
Background	Offset 1.0 and -1.0° in ω from $K\alpha_{1,2}$ maximum
Scan rate, deg min-1	Variable, 2.0 - 5.0
Check reflections	4 remeasured after every 96 reflections; analysis[e] of these data indicated a steady decline in intensity by ca. 9% at the conclusion of data collection. A correction for this effect was applied.
2θ range, deg	4.0 - 60.0
Total reflections measured	1112

Table I. continued...

[a]Unit cell parameters were obtained by least-squares refinement of the setting angles of 41 reflections with $13.0 < 2\theta < 19.6°$.[b] Due to air-sensitivity an experimental density was not determined.[c] Shown by successful refinement to be *C2/c* (see text).[d] Syntex $P2_1$ autodiffractometer equipped with a graphite monochromator and a Syntex LT-1 inert-gas low-temperature delivery system.[e] W. H. Henslee and R. E. Davis, *Acta Crystallogr., Sect. B*, **31**, 1511 (1975).

Table II. Fractional Coordinates and Anisotropic (X 10^4) and Isotropic Thermal Parameters for the Atoms of $(Me_2N)_2SF_2$.[a]

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
S	0	-0.0273 (2)	1/4	76 (1)	167 (4)	50 (1)	0	-19 (1)	0
F	0.0977 (2)	-0.0417 (4)	0.1395 (2)	103 (2)	653 (12)	64 (2)	126 (4)	-5 (2)	-95 (4)
N	0.0965 (2)	0.1542 (4)	0.3133 (2)	51 (2)	203 (9)	44 (2)	-4 (4)	-4 (2)	-10 (3)
C(1)	0.2013 (3)	0.0240 (8)	0.3620 (3)	63 (3)	297 (14)	70 (3)	6 (6)	-12 (3)	0 (6)
C(2)	0.0476 (4)	0.3311 (7)	0.3868 (3)	91 (4)	271 (13)	56 (3)	14 (7)	-6 (3)	-32 (5)

Atom[b]	x	y	z	B, $Å^2$
H (1, 1)	0.264 (3)	0.135 (7)	0.372 (3)	5.2 (10)
H (1, 2)	0.186 (3)	-0.068 (6)	0.428 (3)	4.8 (9)
H (1, 3)	0.232 (3)	-0.103 (7)	0.312 (3)	5.1 (9)
H (2, 1)	0.103 (4)	0.434 (7)	0.394 (3)	6.2 (12)
H (2, 2)	-0.028 (4)	0.410 (7)	0.354 (3)	6.0 (10)
H (2, 3)	0.030 (3)	0.258 (7)	0.464 (3)	5.2 (10)

[a] See Figure 1 for identity of the atoms. Numbers in parentheses throughout the table are the estimated standard deviations in the units of the least significant digits for the corresponding parameter. The anisotropic temperature factor is $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}\ell^2 + 2\beta_{12}hk + 2\beta_{13}h\ell + 2\beta_{23}k\ell)]$. [b] The sequence number of a hydrogen atom corresponds to that of the carbon atom to which it is bound.

Table III. Selected Geometric Parameters for $(Me_2N)_2SF_2$ with Estimated Standard Deviations in Parentheses. Coordinates of primed atoms are related to the coordinates of the corresponding unprimed atoms of Table II by the operation -x, y, ½-z.

Bond Distances (Å)

S-F	1.770 (2)
S-N	1.648 (2)
N-C(1)	1.479 (5)
N-C(2)	1.470 (5)

Bond Angles (deg)

F-S-F'	174.7 (1)
N-S-N'	102.3 (1)
N-S-F	94.1 (1)
N-S-F'	89.2 (1)
S-N-C(1)	110.6 (2)
S-N-C(2)	118.1 (2)
C(1)-N-C(2)	113.1 (3)

Dihedral Angles (deg)

C(1)-N-C(2) and N-S-N' planes 48.2°

N-S-N' and F-S-F' planes 86.9°

C(1)-N-C(2) and F-S-F' planes 105.2°

Table IV. Equatorial Bond Angles (θ_1), and Axial Bond Distances for Sulfuranes

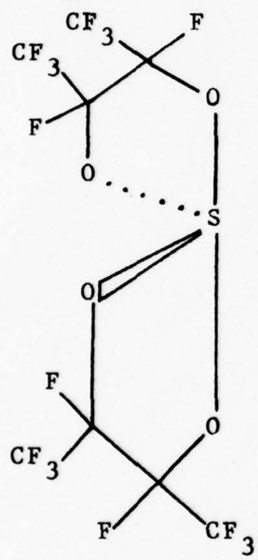
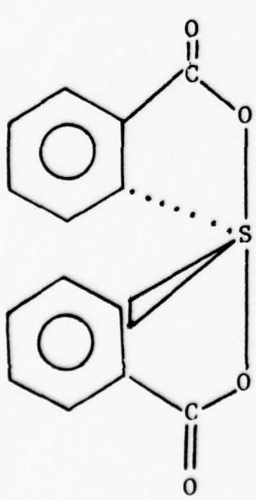
Sulfurane	θ_1 (deg)[a]	Axial Bond Distance (Å)
SF_4	101.6[b]	1.64[b]
$(Me_2N)_2SF_2$	102.3[c]	1.77[c]
$\phi_2S[OC(O)(CF_3)_2]_2$	104.4[d]	1.889, 1.916[d]
	104.6[e]	1.754, 1.756[e]
	107.8[f]	1.83[f]

Table IV continued...

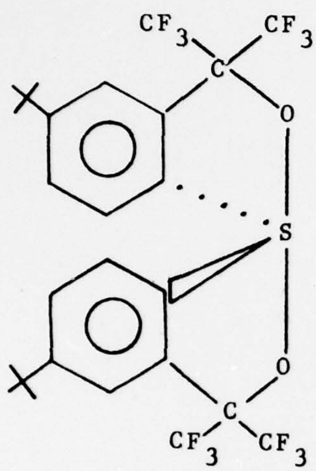
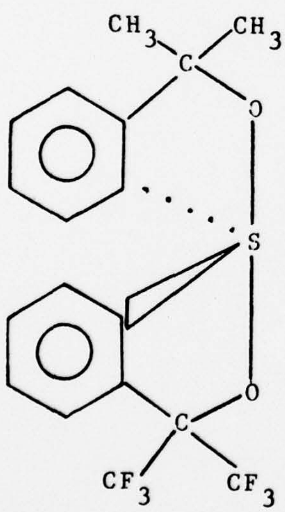
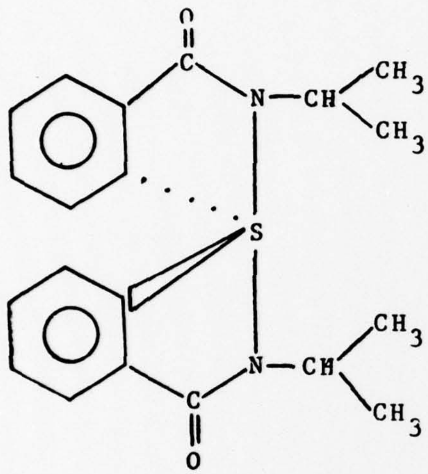
Sulfurane	θ_1 (deg)[a]	Axial Bond Distance (Å)
	107.6, 108.1[g,h]	1.832, 1.819[g,h] 1.831, 1.816[g,h]
	108.1[i]	1.713, 1.955

Table IV continued...

Sulfurane	θ_1 (deg)[a]	Axial Bond Distance (Å)
	104.7[j]	1.897, 1.899
$(ClC_6H_4)_2SCl_2$	108.6[k]	2.259, 2.323

[a] θ_1 is defined in 7

[b] Reference 10

[c] Present work

[d] Reference 13

[e] Reference 8

[f] Reference 5

[g] Reference 6

[h] Two crystallographically independent molecules per unit cell.

[i] Reference 7

[j] Reference 9

[k] Reference 12

SUPPLEMENTARY TABLE. Observed and calculated structure factors for $[(CH_3)_2N]_2SF_2$. The running index is _h_; values of _k_ and _ℓ_ are presented in the first two columns on the left-hand side, the third column is $10|F_o|$, and the right-hand column is $10|F_c|$. Reflections marked with * were not used in least-squares refinement.

K	L	FOBS	FCAL	K	L	FOBS	FCAL	K	L	FOBS	FCAL	K	L	FOBS	FCAL
***	H =	0	***	1	-16	48	85*	3	9	123	126	0	-10	3	7*
0	4	748	741	1	-15	43	54*	3	10	124	126	0	-8	405	405
0	6	50	39*	1	-14	21	38*	3	11	131	135	0	-6	593	576
0	8	474	475	1	-13	3	12*	3	12	3	5*	0	-4	403	386
0	10	236	232	1	-12	81	82	3	13	63	51*	0	-2	729	767
0	12	3	24*	1	-11	158	155	3	14	69	40	0	2	320	309
0	14	98	110	1	-10	227	220	3	15	57	9*	0	4	185	190
0	16	137	144	1	-9	147	148	5	-13	35	14*	0	6	60	55
2	0	796	840	1	-8	276	269	5	-12	72	43	0	8	138	128
2	1	175	182	1	-7	104	97	5	-11	50	22*	0	10	229	238
2	2	459	438	1	-6	163	166	5	-10	73	75	0	12	278	297
2	3	204	198	1	-5	94	84	5	-9	63	74	0	14	196	209
2	4	96	74	1	-4	273	270	5	-8	66	67	0	16	64	51*
2	5	46	29*	1	-3	109	108	5	-7	158	152	2	-16	19	53*
2	6	133	144	1	-1	606	614	5	-6	87	78	2	-15	4	45*
2	7	291	284	1	1	97	89	5	-5	107	95	2	-14	76	94
2	8	443	441	1	2	618	625	5	-4	158	148	2	-13	8	3*
2	9	225	232	1	3	553	533	5	-3	113	122	2	-12	136	136
2	10	332	332	1	4	44	40*	5	-2	168	169	2	-11	58	50*
2	11	76	61	1	5	170	151	5	-1	279	288	2	-10	136	129
2	12	56	66*	1	6	62	49	5	0	139	135	2	-9	301	303
2	13	48	2*	1	7	34	2*	5	1	302	303	2	-8	220	216
2	14	16	8*	1	8	275	273	5	2	128	128	2	-7	276	285
2	15	4	8*	1	9	125	137	5	3	108	118	2	-6	349	353
2	16	64	64*	1	10	323	329	5	4	120	97	2	-5	325	300
4	0	179	166	1	11	109	118	5	5	45	3*	2	-4	285	293
4	1	265	268	1	12	139	153	5	6	100	95	2	-3	505	484
4	2	125	121	1	13	35	49*	5	7	3	37*	2	-2	73	58
4	3	254	253	1	14	71	73	5	8	101	114	2	-1	174	183
4	4	133	116	1	15	68	72	5	9	103	80	2	0	101	102
4	5	86	80	1	16	67	92*	5	10	76	81	2	1	420	400
4	6	198	198	3	-15	4	17*	5	11	97	95	2	2	664	673
4	7	43	26*	3	-14	59	64*	5	12	54	23*	2	3	248	232
4	8	251	249	3	-13	53	15*	5	13	93	95	2	4	575	556
4	9	93	67	3	-12	52	53*	7	-8	30	18*	2	5	235	210
4	10	171	168	3	-11	72	58	7	-7	84	88	2	6	180	182
4	11	109	121	3	-10	220	214	7	-6	4	22*	2	7	189	180
4	12	60	43*	3	-9	156	157	7	-5	101	109	2	8	152	158
4	13	60	47*	3	-8	420	418	7	-4	87	76	2	9	3	55*
4	14	33	1*	3	-7	210	204	7	-3	79	74	2	10	220	225
6	0	219	225	3	-6	233	227	7	-2	82	90	2	11	20	5*
6	1	142	153	3	-5	93	97	7	-1	4	36*	2	12	129	140
6	2	113	118	3	-4	150	127	7	0	82	67	2	13	3	18*
6	3	142	139	3	-3	75	63	7	1	80	101	2	14	4	50*
6	4	38	2*	3	-2	96	94	7	2	50	37*	2	15	4	3*
6	5	106	118	3	-1	97	117	7	3	159	160	2	16	53	50*
6	6	54	46*	3	0	463	456	7	4	37	19*	4	-14	80	87
6	7	70	75	3	1	333	313	7	5	84	103	4	-13	44	13*
6	8	86	62	3	2	172	175	7	6	4	3*	4	-12	116	128
6	9	48	52*	3	3	255	254	7	7	59	59*	4	-11	35	22*
6	10	4	5*	3	4	79	91	7	8	4	2*	4	-10	91	85
6	11	62	64*	3	5	77	64	***	H =	2	***	4	-9	69	86
8	0	4	13*	3	6	406	406	0	-16	120	123	4	-8	87	79
***	H =	1	***	3	7	3	12*	0	-14	103	98	4	-7	165	167
1	-17	50	9*	3	8	421	424	0	-12	70	58	4	-6	193	187

K	L	FOBS	FCAL	K	L	FOBS	FCAL	K	L	FOBS	FCAL	K	L	FOBS	FCAL
4	-5	176	187	1	-5	70	51	5	-11	4	16*	0	14	120	133
4	-4	182	183	1	-4	704	700	5	-10	39	19*	0	16	4	20*
4	-3	137	136	1	-3	193	184	5	-9	124	115	2	-16	54	56*
4	-2	33	28*	1	-2	322	309	5	-8	47	18*	2	-15	35	87*
4	-1	137	135	1	-1	340	326	5	-7	174	179	2	-14	130	125
4	0	66	67	1	0	161	137	5	-6	157	155	2	-13	43	38*
4	1	283	285	1	1	401	384	5	-5	124	134	2	-12	106	111
4	2	268	267	1	2	953	989	5	-4	204	208	2	-11	3	11*
4	3	240	235	1	3	655	619	5	-3	159	168	2	-10	3	3*
4	4	349	341	1	4	692	663	5	-2	76	70	2	-9	59	58*
4	5	27	53*	1	5	149	140	5	-1	200	212	2	-8	100	92
4	6	218	207	1	6	58	42	5	0	57	41*	2	-7	3	4*
4	7	64	55	1	7	71	44	5	1	110	107	2	-6	382	380
4	8	123	114	1	8	41	16*	5	2	177	170	2	-5	125	95
4	9	157	157	1	9	46	1*	5	3	44	3*	2	-4	364	343
4	10	85	95	1	10	322	328	5	4	155	150	2	-3	2	16*
4	11	176	187	1	11	42	3*	5	5	47	63*	2	-2	2	94*
4	12	33	52*	1	12	309	307	5	6	3	45*	2	-1	212	206
4	13	14	65*	1	13	21	48*	5	7	136	142	2	0	172	161
4	14	4	17*	1	14	97	103	5	8	72	60	2	1	281	261
6	-11	36	12*	1	15	28	3*	5	9	103	118	2	2	805	802
6	-10	31	15*	1	16	22	20*	5	10	125	107	2	3	365	322
6	-9	95	70	3	-15	27	46*	5	11	82	104	2	4	526	516
6	-8	56	40*	3	-14	144	151	5	12	22	72*	2	5	307	294
6	-7	139	135	3	-13	4	23*	7	-7	14	46*	2	6	3	47*
6	-6	85	81	3	-12	77	90	7	-6	81	79	2	7	3	18*
6	-5	124	115	3	-11	3	21*	7	-5	60	71*	2	8	29	45*
6	-4	108	118	3	-10	46	59*	7	-4	85	96	2	9	153	156
6	-3	93	90	3	-9	130	119	7	-3	56	75*	2	10	213	207
6	-2	151	164	3	-8	182	164	7	-2	4	40*	2	11	38	39*
6	-1	131	152	3	-7	161	180	7	-1	72	85	2	12	160	164
6	0	111	113	3	-6	218	209	7	0	4	5*	2	13	97	96
6	1	179	181	3	-5	78	72	7	1	111	127	2	14	78	70
6	2	47	1*	3	-4	65	66	7	2	37	16*	2	15	68	70*
6	3	111	116	3	-3	72	51	7	3	124	129	4	-14	81	105
6	4	3	2*	3	-2	2	18*	7	4	4	17*	4	-13	36	41*
6	5	80	55	3	-1	98	82	7	5	87	86	4	-12	94	107
6	6	72	69	3	0	230	229	7	6	4	3*	4	-11	7	27*
6	7	79	63	3	1	266	263	7	7	79	79	4	-10	34	15*
6	8	44	51*	3	2	499	468	*** H = 4 ***				4	-9	28	20*
6	9	102	104	3	3	267	266	0	-16	81	80	4	-8	3	16*
6	10	81	23	3	4	528	530	0	-14	32	61*	4	-7	151	163
6	11	88	107	3	5	157	146	0	-12	3	53*	4	-6	159	150
*** H = 3 ***				3	6	278	279	0	-10	53	45*	4	-5	284	299
1	-16	49	65*	3	7	60	57*	0	-8	423	413	4	-4	153	147
1	-15	4	9*	3	8	31	31*	0	-6	695	676	4	-3	161	153
1	-14	115	116	3	9	68	49	0	-4	444	430	4	-2	48	54*
1	-13	67	76	3	10	54	33*	0	-2	45	7*	4	-1	70	55
1	-12	74	53	3	11	70	72	0	0	2	61*	4	0	197	186
1	-11	118	102	3	12	124	121	0	2	354	335	4	1	37	61*
1	-10	58	65*	3	13	32	66*	0	4	509	504	4	2	372	374
1	-9	43	21*	3	14	72	86	0	6	256	250	4	3	183	169
1	-8	230	212	3	15	43	32*	0	8	113	117	4	4	265	264
1	-7	44	21*	5	-13	52	26*	0	10	254	264	4	5	102	112
1	-6	824	817	5	-12	57	68*	0	12	311	321	4	6	59	33*

K	L	FOBS	FCAL	K	L	FOBS	FCAL	K	L	FOBS	FCAL	K	L	FOBS	FCAL
4	7	100	101	1	10	198	208	5	7	147	159	2	6	304	304
4	8	3	39*	1	11	37	7*	5	8	118	122	2	7	56	40*
4	9	138	134	1	12	128	122	5	9	78	62	2	8	192	199
4	10	101	118	1	13	52	16*	5	10	85	99	2	9	73	72
4	11	98	107	1	14	80	78	5	11	82	79	2	10	88	60
4	12	60	85*	1	15	66	69*	7	-6	52	50*	2	11	90	61
4	13	69	62	3	-15	30	67*	7	-5	105	105	2	12	71	68
6	-10	23	39*	3	-14	52	72*	7	-4	38	20*	2	13	116	120
6	-9	66	42*	3	-13	85	67	7	-3	80	90	2	14	133	135
6	-8	68	87	3	-12	40	72*	7	-2	4	2*	4	-13	69	86*
6	-7	98	99	3	-11	26	15*	7	-1	62	60*	4	-12	32	73*
6	-6	18	83*	3	-10	99	88	7	0	56	11*	4	-11	46	47*
6	-5	141	138	3	-9	26	38*	7	1	94	104	4	-10	70	52
6	-4	78	79	3	-8	57	25*	7	2	38	18*	4	-9	3	3*
6	-3	134	132	3	-7	94	103	7	3	99	129	4	-8	54	10*
6	-2	104	89	3	-6	46	27*	7	4	4	18*	4	-7	116	129
6	-1	88	91	3	-5	212	235	7	5	51	82*	4	-6	64	14
6	0	18	37*	3	-4	167	161	*** H = 6 ***				4	-5	199	212
6	1	61	56*	3	-3	111	123	0	-16	4	2*	4	-4	156	164
6	2	65	14	3	-2	431	433	0	-14	4	21*	4	-3	3	33*
6	3	89	90	3	-1	3	15*	0	-12	153	150	4	-2	285	291
6	4	41	28*	3	0	387	376	0	-10	273	260	4	-1	101	82
6	5	124	136	3	1	54	57*	0	-8	120	117	4	0	231	231
6	6	73	80	3	2	214	198	0	-6	63	76	4	1	100	83
6	7	110	116	3	3	215	212	0	-4	392	382	4	2	74	87
6	8	41	75*	3	4	220	233	0	-2	439	448	4	3	192	186
6	9	69	78	3	5	220	207	0	0	197	182	4	4	61	56
6	10	58	72*	3	6	194	185	0	2	280	262	4	5	131	141
*** H = 5 ***				3	7	64	40	0	4	536	535	4	6	102	103
1	-16	4	12*	3	8	37	38*	0	6	688	705	4	7	122	132
1	-15	78	49	3	9	51	7*	0	8	269	274	4	8	96	108
1	-14	64	69*	3	10	43	37*	0	10	3	29*	4	9	113	125
1	-13	33	84*	3	11	82	60	0	12	99	93	4	10	45	69*
1	-12	113	109	3	12	128	126	0	14	109	107	4	11	48	56*
1	-11	49	26*	3	13	77	90	2	-15	43	57*	4	12	76	65
1	-10	101	83	3	14	106	118	2	-14	4	56*	6	-9	4	43*
1	-9	151	161	5	-12	51	72*	2	-13	54	15*	6	-8	67	68*
1	-8	161	152	5	-11	67	41*	2	-12	125	139	6	-7	96	82
1	-7	45	25*	5	-10	74	70	2	-11	56	33*	6	-6	4	15*
1	-6	415	397	5	-9	97	79	2	-10	92	83	6	-5	93	95
1	-5	176	171	5	-8	36	39*	2	-9	50	61*	6	-4	14	19*
1	-4	443	431	5	-7	117	125	2	-8	44	34*	6	-3	50	63*
1	-3	48	48*	5	-6	56	62*	2	-7	39	23*	6	-2	91	78
1	-2	124	133	5	-5	158	172	2	-6	77	74	6	-1	3	46*
1	-1	31	17*	5	-4	84	98	2	-5	124	136	6	0	35	18*
1	0	71	58	5	-3	148	148	2	-4	393	383	6	1	61	83*
1	1	396	356	5	-2	116	96	2	-3	272	278	6	2	49	14*
1	2	234	218	5	-1	17	8*	2	-2	435	434	6	3	123	132
1	3	305	275	5	0	130	114	2	-1	213	203	6	4	76	81
1	4	358	358	5	1	71	50	2	0	213	204	6	5	102	119
1	5	59	40	5	2	114	116	2	1	57	13	6	6	126	133
1	6	348	338	5	3	91	102	2	2	99	77	6	7	50	78*
1	7	86	104	5	4	28	56*	2	3	90	81	6	8	31	72*
1	8	269	263	5	5	232	236	2	4	183	184	*** H = 7 ***			
1	9	28	31*	5	6	64	66	2	5	118	116	1	-15	4	19*

K	L	FOBS	FCAL
1	-14	19	69*
1	-13	42	26*
1	-12	169	167
1	-11	52	58*
1	-10	128	128
1	-9	88	98
1	-8	49	16*
1	-7	3	39*
1	-6	80	69
1	-5	179	171
1	-4	300	298
1	-3	141	151
1	-2	319	312
1	-1	105	95
1	0	16	22*
1	1	98	90
1	2	9	26*
1	3	67	68
1	4	374	362
1	5	126	121
1	6	483	468
1	7	3	10*
1	8	232	232
1	9	78	74
1	10	3	21*
1	11	42	29*
1	12	7	39*
1	13	43	32*
1	14	99	114
3	-14	4	8*
3	-13	4	42*
3	-12	70	47
3	-11	3	25*
3	-10	86	92
3	-9	28	1*
3	-8	55	15*
3	-7	10	28*
3	-6	23	9*
3	-5	110	104
3	-4	322	324
3	-3	155	164
3	-2	465	471
3	-1	91	119
3	0	105	112
3	1	47	21*
3	2	71	69
3	3	54	50*
3	4	168	157
3	5	88	90
3	6	200	199
3	7	106	98
3	8	73	45
3	9	80	85
3	10	71	57
3	11	53	55*
3	12	149	161
3	13	4	37*
5	-11	84	100
5	-10	82	62
5	-9	95	86
5	-8	51	28*
5	-7	60	18*
5	-6	16	32*
5	-5	44	17*
5	-4	95	74
5	-3	42	72*
5	-2	79	87
5	-1	95	65
5	0	95	78
5	1	86	58
5	2	77	67
5	3	132	130
5	4	95	81
5	5	167	178
5	6	111	110
5	7	120	125
5	8	74	86
5	9	75	62
5	10	4	38*
7	-3	18	60*
7	-2	4	5*
7	-1	58	88*
7	0	46	22*
7	1	90	106
7	2	4	40*
*** H =	8 ***		
0	-14	67	48*
0	-12	129	138
0	-10	152	176
0	-8	27	30*
0	-6	54	60*
0	-4	130	123
0	-2	268	267
0	0	105	127
0	2	201	203
0	4	427	430
0	6	324	328
0	8	93	89
0	10	7	44*
0	12	73	90
0	14	38	65*
2	-14	27	26*
2	-13	23	15*
2	-12	64	54*
2	-11	30	31*
2	-10	3	54*
2	-9	3	6*
2	-8	83	74
2	-7	46	33*
2	-6	124	131
2	-5	125	119
2	-4	192	194
2	-3	282	295
2	-2	212	202
2	-1	128	132
2	0	179	184
2	1	96	124
2	2	142	132
2	3	56	86*
2	4	118	111
2	5	48	43*
2	6	171	163
2	7	70	67
2	8	163	157
2	9	87	69
2	10	93	116
2	11	99	91
2	12	112	105
2	13	68	62*
4	-12	50	6*
4	-11	94	81
4	-10	4	29*
4	-9	4	32*
4	-8	92	86
4	-7	37	4*
4	-6	85	113
4	-5	48	2*
4	-4	95	107
4	-3	3	52*
4	-2	130	118
4	-1	139	128
4	0	122	115
4	1	107	130
4	2	77	76
4	3	65	61
4	4	53	49*
4	5	104	95
4	6	59	65*
4	7	162	161
4	8	90	100
4	9	63	85*
4	10	99	106
4	11	33	19*
6	-7	58	12*
6	-6	21	15*
6	-5	55	1*
6	-4	39	1*
6	-3	51	69*
6	-2	4	23*
6	-1	111	116
6	0	56	27*
6	1	91	84
6	2	78	77
6	3	83	59
6	4	113	121
6	5	62	79*
6	6	55	66*
*** H =	9 ***		
1	-14	61	41*
1	-13	51	4*
1	-12	74	43
1	-11	49	15*
1	-10	93	84
1	-9	3	14*
1	-8	121	116
1	-7	65	77
1	-6	58	31*
1	-5	98	89
1	-4	49	22*
1	-3	48	59*
1	-2	113	159
1	-1	39	41*
1	0	360	379
1	1	15	28*
1	2	271	280
1	3	126	138
1	4	85	69
1	5	19	37*
1	6	96	94
1	7	122	118
1	8	184	182
1	9	97	101
1	10	111	130
1	11	7	21*
1	12	46	48*
1	13	4	16*
3	-12	4	16*
3	-11	66	58*
3	-10	62	89*
3	-9	40	41*
3	-8	121	139
3	-7	41	16*
3	-6	93	113
3	-5	72	29
3	-4	93	104
3	-3	124	144
3	-2	98	112
3	-1	120	123
3	0	98	92
3	1	25	2*
3	2	80	82
3	3	3	10*
3	4	71	65
3	5	89	80
3	6	68	57
3	7	104	118
3	8	116	128
3	9	69	73
3	10	175	186
3	11	41	30*

K	L	FOBS	FCAL
5	-9	4	35*
5	-8	72	71
5	-7	87	58
5	-6	9	64*
5	-5	12	13*
5	-4	4	8*
5	-3	92	105
5	-2	3	4*
5	-1	111	114
5	0	102	108
5	1	95	76
5	2	112	121
5	3	103	107
5	4	48	50*
5	5	120	122
5	6	82	36
5	7	46	56*
5	8	35	62*
*** H = 10 ***			
0	-12	65	41*
0	-10	98	96
0	-8	38	43*
0	-6	22	53*
0	-4	3	9*
0	-2	227	242
0	0	353	363
0	2	252	248
0	4	84	61
0	6	3	19*
0	8	120	113
0	10	134	137
0	12	68	60*
2	-12	11	2*
2	-11	71	60
2	-10	67	78
2	-9	50	64*
2	-8	152	159
2	-7	27	28*
2	-6	92	79
2	-5	78	62
2	-4	45	26*
2	-3	97	80
2	-2	97	96
2	-1	3	19*
2	0	252	256
2	1	74	77
2	2	126	121
2	3	20	11*
2	4	3	22*
2	5	90	78
2	6	96	97
2	7	77	73
2	8	212	215
2	9	85	86
2	10	129	142

K	L	FOBS	FCAL
2	11	71	76
4	-10	41	47*
4	-9	4	21*
4	-8	108	133
4	-7	48	10*
4	-6	72	84
4	-5	3	4*
4	-4	3	15*
4	-3	3	40*
4	-2	3	27*
4	-1	123	128
4	0	104	109
4	1	118	130
4	2	61	67*
4	3	58	60*
4	4	3	14*
4	5	40	47*
4	6	73	80
4	7	4	52*
4	8	140	143
4	9	64	21*
6	-3	56	52*
6	-2	66	35*
6	-1	72	68
6	0	27	64*
6	1	47	65*
*** H = 11 ***			
1	-12	62	6*
1	-11	4	16*
1	-10	16	41*
1	-9	45	70*
1	-8	55	68*
1	-7	60	70*
1	-6	3	25*
1	-5	37	22*
1	-4	3	30*
1	-3	57	67*
1	-2	166	179
1	-1	3	4*
1	0	246	250
1	1	51	11*
1	2	87	105
1	3	3	20*
1	4	18	13*
1	5	3	42*
1	6	93	91
1	7	96	98
1	8	141	132
1	9	4	54*
1	10	77	74
1	11	47	2*
3	-10	75	75
3	-9	51	63*
3	-8	64	83*
3	-7	4	42*

K	L	FOBS	FCAL
3	-6	60	64*
3	-5	12	7*
3	-4	50	45*
3	-3	3	1*
3	-2	3	19*
3	-1	47	54*
3	0	3	17*
3	1	66	77
3	2	71	68
3	3	41	57*
3	4	104	113
3	5	33	30*
3	6	130	124
3	7	83	40
3	8	122	125
3	9	89	59
5	-6	34	42*
5	-5	36	32*
5	-4	62	20*
5	-3	4	49*
5	-2	54	35*
5	-1	69	75
5	0	75	67
5	1	105	106
5	2	4	47*
5	3	76	82
5	4	26	31*
*** H = 12 ***			
0	-10	4	10*
0	-8	4	5*
0	-6	92	88
0	-4	156	165
0	-2	124	122
0	0	74	72
0	2	104	120
0	4	113	103
0	6	34	12*
0	8	46	21*
0	10	94	99
2	-10	4	21*
2	-9	51	57*
2	-8	37	56*
2	-7	50	27*
2	-6	76	73
2	-5	38	22*
2	-4	50	48*
2	-3	38	56*
2	-2	48	29*
2	-1	19	26*
2	0	69	48
2	1	55	61*
2	2	67	88
2	3	89	94
2	4	127	131
2	5	15	47*

K	L	FOBS	FCAL
2	6	137	124
2	7	23	34*
2	8	60	65*
2	9	38	54*
4	-7	41	55*
4	-6	50	44*
4	-5	4	4*
4	-4	30	29*
4	-3	16	13*
4	-2	4	7*
4	-1	95	101
4	0	4	7*
4	1	97	100
4	2	63	53*
4	3	4	8*
4	4	114	108
4	5	4	19*
*** H = 13 ***			
1	-9	52	36*
1	-8	36	44*
1	-7	4	7*
1	-6	83	106
1	-5	23	59*
1	-4	47	90*
1	-3	57	48*
1	-2	3	32*
1	-1	36	27*
1	0	53	36*
1	1	76	46
1	2	97	95
1	3	31	33*
1	4	107	99
1	5	53	53*
1	6	4	37*
1	7	4	40*
1	8	4	21*
3	-7	32	25*
3	-6	50	53*
3	-5	37	5*
3	-4	41	40*
3	-3	4	1*
3	-2	43	40*
3	-1	4	38*
3	0	16	3*
3	1	46	57*
3	2	124	141
3	3	4	40*
3	4	151	158
3	5	56	24*
3	6	27	51*
*** H = 14 ***			
0	-8	4	48*
0	-6	107	120
0	-4	86	83
0	-2	19	10*

K	L	FOBS	FCAL	K	L	FOBS	FCAL	K	L	FOBS	FCAL	K	L	FOBS	FCAL
0	0	4	13*	2	-4	69	17	2	2	91	101	1	-3	4	9*
0	2	60	88*	2	-3	41	16*	2	3	43	83*	1	-2	4	37*
0	4	4	44*	2	-2	28	9*	2	4	70	81	1	-1	4	33*
0	6	4	4*	2	-1	21	3*	2	5	63	32*	1	0	67	32
2	-6	4	42*	2	0	53	58*	*** H = 15 ***				1	1	4	33*
2	-5	23	3*	2	1	47	66*	1	-4	4	29*	1	2	4	16*

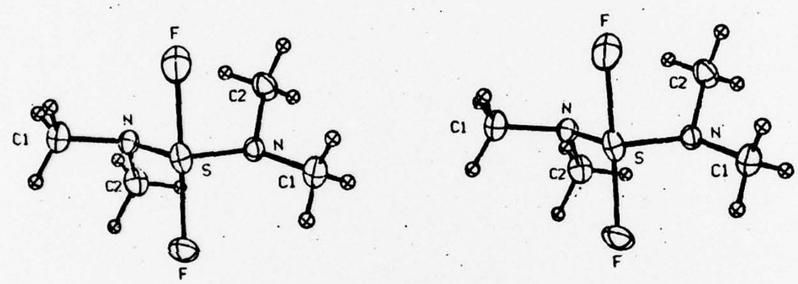


Figure 1. Stereoview of the $(Me_2N)_2SF_2$ molecule, illustrating the atom numbering scheme. Nonhydrogen atoms are shown as ellipsoids of 30% probability and hydrogen atoms as spheres of radius 0.1 Å.

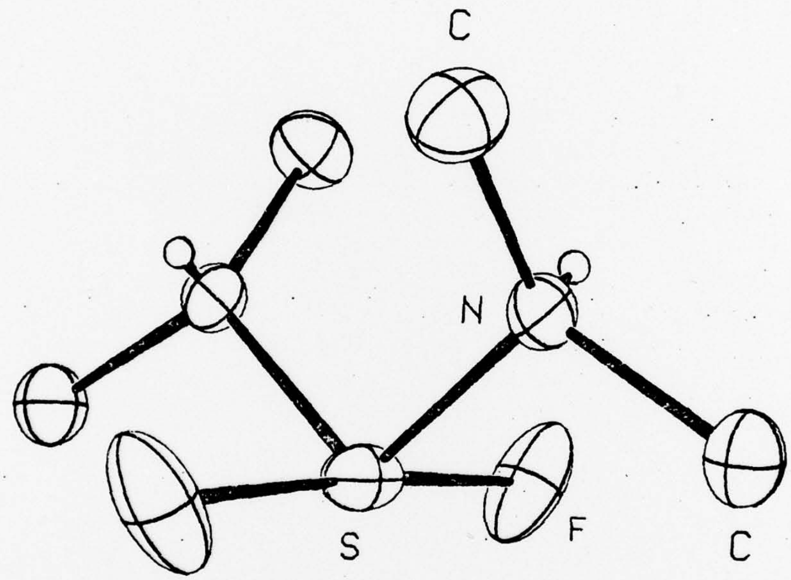


Figure 2. A view of the $(Me_2N)_2SF_2$ molecule showing the idealized dispositions of the lone pairs of electrons of the nitrogen atoms. The lone pairs, indicated as small blank spheres, have been placed along the axes which pass through the N atoms and are normal to the S, C, C planes.

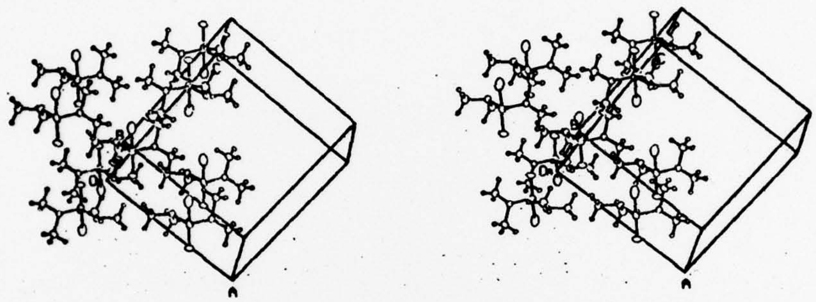


Figure 3. Stereoview of the immediate environment of a $(Me_2N)_2SF_2$ molecule. Each molecule is surrounded by eight other molecules at nonhydrogen contact distances of 3.6-3.85 Å in an approximately square antiprismatic arrangement of molecules. Nonhydrogen atoms are shown as ellipsoids of 30% probability and hydrogen atoms as spheres of radius 0.1 Å.

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